REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*
05/06/2002

2. REPORT DATE
FINAL REPORT

3. DATES COVERED *(From - To)*
06/01/1996–09/30/00

4. TITLE AND SUBTITLE
Total Quality Optimal Fabrication of Composite Materials via Liquid Molding

5a. CONTRACT NUMBER
N00014-96-1-0726

5b. GRANT NUMBER
N00014-96-1-0726

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER
N00014-96-1-0726

5e. TASK NUMBER

5f. WORK UNIT NUMBER

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
ONR 332, Materials S&T
800 North Quincy Street
Arlington, VA

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSORING/MONITORING AGENCY REPORT NUMBER

12. DISTRIBUTION AVAILABILITY STATEMENT
Distribution: Unlimited

20020619 011

13. SUPPLEMENTARY NOTES

14. ABSTRACT
Liquid composite molding techniques potentially offer the capability to fabricate high-performance composite products of complex shapes at high throughputs. Strong parameter uncertainties and variabilities inherent in the process have remained a fundamental impediment to a complete realization of the process potential and the widespread use of the process in commercial and DoD applications. The overall research objective is to overcome the fundamental impediments and significantly advance the processing science and technology, leading to capabilities for rapid, robust and affordable manufacturing of composites.

15. SUBJECT TERMS
Liquid Composite Molding, Intelligent Control, Model predictive control, Total quality manufacturing, Preform permeability, Nondestructive evaluation

16. SECURITY CLASSIFICATION OF: a. REPORT	b. ABSTRACT	c. THIS PAGE	17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
U	U	U	UU	9	James J. Kelly
					19b. TELEPONE NUMBER *(Include area code)* (703) 696-0688; kellyj@onr.navy.mil

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5a. CONTRACT NUMBER. Enter all contract numbers as they appear in the report, e.g. F33615-86-C-5169.

5b. GRANT NUMBER. Enter all grant numbers as they apperar in the report, e.g. 1F665702D1257.

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5d. PROJECT NUMBER. Enter al project numbers as they appear in the report, e.g. 1F665702D1257; ILIR.

5e. TASK NUMBER. Enter all task numbers as they appear in the report, e.g. 05; RF0330201; T4112.

5f. WORK UNIT NUMBER. Enter all work unit numbers as they appear in the repor, e.g. 001; AFAPL30480105.

6. AUTHOR(S). Enter name(s) of person(s) responsible for writing the report, performing the research, or credited with the content of the report. The form of entry is the last name, first name, middle initial, and additional qualifiers separated by commas, e.g. Smith, Richard, Jr.

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8. PERFORMING ORGANIZATION REPORT NUMBER. Enter all unique alphanumeric report numbers assigned by the performing organization, e.g. BRL-1234; AFWL-TR-85-4017-Vol-21-PT-2.

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14. ABSTRACT. A brief (approximately 200 words) factual summary of the most significant information.

15. SUBJECT TERMS. Key words or phrases identifying major concepts in the report.

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17. LIMITATION OF ABSTRACT. This block must be completed to assign a distribution limitation to the abstract. Enter UU (Unclassified Unlimited) or SAR (Same as Report). An entry in this block is necessary if the abstract is to be limited.

Total Quality Optimal Fabrication of Composite Materials via Liquid Molding
and
Intelligent Simulation-Assisted Liquid Composite Molding

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Contract Numbers: **N00014-96-1-0726 and N00014-97-1-0730**

FINAL REPORT – Part I

ONR PROGRAM OFFICER: James J. Kelly

LONG-TERM RESEARCH OBJECTIVE:
Liquid composite molding techniques potentially offer the capability to fabricate high-performance composite products of complex shapes at high throughputs. Strong parameter uncertainties and variabilities inherent in the process have remained a fundamental impediment to a complete realization of the process potential and the widespread use of the process in commercial and DoD applications. The overall research objective is to overcome the fundamental impediments and significantly advance the processing science and technology, leading to capabilities for rapid, robust and affordable manufacturing of composites.

S&T OBJECTIVES:
- To identify and characterize the source and nature of the process parameter uncertainties,
- To develop a science-based framework for stochastic analysis and optimization of the process; to identify robust processing regimes that minimize process variabilities,
- To devise and demonstrate a robust, real-time, optimal process control framework with the control decisions derived from process physics rather from empirical heuristics, and
- To explore novel strategies for accelerating the resin flow and cure during the process.

APPROACH:
Detailed experiments were conducted on a laboratory scale process toward identifying the critical parameter uncertainties, and obtaining quantification of the uncertainty information. A stochastic analysis framework was developed wherein process simulation models were used to explore the interactive effects of the parameter uncertainties on the resulting process and product quality variabilities. Based on the stochastic analyses, processing regimes that minimize the output variabilities were developed for a wide range of process and material parameters, and quality considerations. Implementation of the simulation models for real-time process control requires that the simulation time scales be much less than the processing time. This was achieved through the use of neural networks trained using the simulation models, which also permitted optimal control in real-time. Resistive heating of conductive elements embedded within the composite layup was explored as a means of accelerating the cure process (thereby reducing cycle time). Additionally, fundamental studies were conducted on issues pertaining to resin permeation through porous preforms, including accurate permeability modeling,

nondestructive preform permeability mapping, and ultrasonic insonation induced flow enhancement during mold filling step of liquid molding.

S&T COMPLETED:

A stochastic modeling framework was developed during the Program was used to investigate liquid composite molding processes in detail. The investigations covered a wide range of parameters for several processing variants, including isothermal mold filling (as in conventional RTM and VARTM) and nonisothermal mold filling (processes such as RIM/SRIM), and curing of composites. Optimum processing maps were derived from the studies in a generic form applicable to several resin classes and fiber architectures.

Experimental runs were conducted on a laboratory-scale process with the objective of extensively characterizing the parameter uncertainty distributions, and of demonstrating the robust processing regimes obtained theoretically. Uncertainty distributions for parameters such as the preform permeability were established as a function of the fiber architecture (plain weave, stitched mat, etc.) and the fiber volume fraction in the preform. The stochastic model was demonstrated to mimic the variabilities inherent in typical processing, and the optimum conditions obtained from the stochastic optimization were also confirmed experimentally.

An intelligent simulation-based framework for real-time control of the mold-filling step of liquid molding processes was developed. The framework incorporates a process simulator based on artificial neural networks trained using physical models, a real-time fuzzy-logic based parameter estimator for the preform permeability (which exhibits considerable spatially and from run-to-run variabilities), and a simulated annealing based optimizer to obtain optimal values of the control parameters on-the-fly. The intelligent model-predictive controller was demonstrated to produce reliable control on a prototype process, for homogeneous and heterogeneous preforms alike.

Resistive curing of composites using embedded conductive fibers was investigated in detail theoretically and experimentally to identify processing envelopes and optimum configurations (number and positioning of the heating elements and the power densities). The technique was shown to be a viable approach to minimizing the cycle time, especially in the fabrication of thick-section composites, as in many Naval applications. Furthermore, a novel idea of using current cycles in addition to cure temperature cycle was explored, and optimum cure temperature and current cycles, as well as optimum number and placement of the resistive heating mats were derived from the studies.

The Program also included fundamental studies on preform permeability modeling in which fractal geometry was used to describe the preform microstructure, and the fractal description was related to the overall preform permeability. Furthermore, a nondestructive method was developed to correlate preform permeability to ultrasound attenuation characteristics of preforms. Toward improving the flow resin through low-permeability regions in the preform, a novel method of ultrasonic insonation assisted flow enhancement was developed.

IMPACT/NAVY RELEVANCE OF THE RESEARCH:

Designing processes for quality fabrication in a reliable manner is of much significance to the composites industry at large, and to many Naval applications. The total quality manufacturing tool and the intelligent process control framework developed as part of the ONR-funded programs will benefit the Navy and its contractors and the commercial industry alike in enabling cost-effective fabrication of high-quality and high-performance composites. The accelerated curing strategy will be especially valuable to the fabrication of thick-section

composites found in many Naval applications (e.g. submarine hulls). The resistive curing technology also presents opportunities in composite repair applications. The studies on issues pertaining to flow in liquid molding processes will lead to better insertion of the processes for fabricating composite structures for Naval and other DoD applications.

OTHER SPONSORED SCIENCE & TECHNOLOGY:

"Processing Fundamentals and Process Modification for Rapid Liquid Molding of High-Performance Composites," $224,460, *National Science Foundation*, 10/1/1995–9/30/1999. *Objective: To investigate the fundamentals of resin flow and cure during liquid molding processes*

"Development of an Automated Precisely-metered Injection System for Resin Transfer Molding," $10,000, *National Science Foundation*, 10/1/1997–9/30/1999. *Objective: To develop an approach and device for precisely mixing resin & catalysts for on-line process control applications.*

"Raman Spectrometer for Optoelectronic and Structural Materials Applications," $150,000, *Office of Naval Research – DURIP Program*, 1/1998 -1/1999 (One of 5 co-PIs). *Objective: To investigate behavior and properties of advanced materials including composites.*

"Rheometers for Materials Research," $231,000 *Army Research Office – DURIP Program*, 5/25/1999–5/24/2000 (One of 5 co-PIs) *Objective: To characterize temperature and shear-dependent rheology of polymers and composites*

"Advanced Tribometer and Non-Contact Profilometer for Structural, Magnetic and Bio-Materials Applications," $176,796, *Office of Naval Research-DURIP Program*, 2/1/2000–1/31/2001 (One of 9 co-PIs) *Objective: To characterize surface characteristics and polymers and composites.*

"Exploratory Investigations on a Novel Process for the Fabrication of Multiscale Reinforced Polymer Composites," $70,000, National Science Foundation, 9/1/1999–8/31/2001. *Objective: To develop a technique for synthesis of a novel class of reinforcing fibers with hierarchically branched morphology.*

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Contract Numbers: **N00014-96-1-0726 and N00014-97-1-0730**

FINAL REPORT—Part 2

ONR Program Officer: James J. Kelly

SUBCONTRACTORS None

PRODUCTIVITY

__Technology Transfer:__

The research and development from the ONR-funded efforts so far have attracted much interest from the industries and the composites community. Several industries, including Ford Scientific Research Laboratories and Sikorsky Aircraft, have expressed interest in adopting the methodologies and tools developed for robust design of liquid molding processes in the face of parameter uncertainties. The research on intelligent simulation-based process control is of direct relevance to the MANTECH program at Sikorsky aircraft, who has expressed considerable interest in the technologies developed by the PI's group under ONR-funding. Discussions for a technology transfer are underway with Sikorsky and other units of United Technologies Corporation (Hamilton Standard and Dow UT). Apart from the industries, the Polymers Division at the National Institute of Standards and Technology (NIST) is also interested in the outcome of the ONR-funded efforts. Details of collaboration with NIST are also presently being worked out.

SUMMARY OF PUBLICATIONS/PATENTS/PRESENTATIONS/HONORS/PARTICIPANTS FOR CONTRACT NUMBERS: N00014-96-1-0726, N00014-97-1-0730

(a) Number of papers submitted to refereed journals but not yet published	0
(b) Number of papers published in refereed journals	14
(c) Number of Books or Chapters submitted but not yet published	1
(d) Number of Books or Chapters published	6
(e) Number of Printed Technical Reports and Non-refereed papers	12
(f) Number of Patents filed	0
(g) Number of Patents granted	0
(h) Number of invited presentations at Workshops or Professional Society Meetings	7
(i) Number of contributed presentations at Workshops or Professional Society Meetings	6
(j) Honors/Awards/Prizes for Contract/Grant Employees	2
(k) Number of Graduate Students & Post-Docs Supported at least 25%on contract grants	2

Grad Students:	TOTAL		5
		Female	0
		Minority	0
Post Doc:	TOTAL		0
		Female	0
		Minority	0

Number of Female or Minority PI's or co-PI's:

New Female	0
Continuing Female	0
New Minority	0
Continuing Minority	0

(l) Other funding (list attached)

DETAILS ON FOLLOWING PAGES

(b) Journal Publications Appearing in Print:

- S. K. Padmanabhan and R. Pitchumani, "Stochastic Analysis of Isothermal Cure of Resin Systems," *Polymer Composites,* Vol. 20, No. 1, pp. 72–85, 1999
- B. Ramakrishnan and R. Pitchumani, "A Fractal Geometry Model for Evaluating Permeabilities of Porous Preforms Used in Liquid Composite Molding," *International Journal of Heat and Mass Transfer,* Vol. 42, No. 12, pp. 2219–2232, 1999
- S. K. Padmanabhan and R. Pitchumani, "Stochastic Modeling of Nonisothermal Flow During Resin Transfer Molding Processes," *International Journal of Heat and Mass Transfer,* Vol. 42, No. 16, pp. 3057–3070, 1999
- N. Rai and R. Pitchumani, "Neural Network-based Optimal Curing of Composite Materials," *Journal of Materials Processing and Manufacturing Science,* Vol. 6, No. 1, pp. 39–62, 1997.
- L. Zhu and R. Pitchumani, "Processing Envelopes for Supplemental Internal Resistive Heating During Composites Cure," *Journal of Reinforced Plastics and Composites,* 18(13), 1242–1253, 1999
- B. Ramakrishnan, L. Zhu, and R. Pitchumani, "Curing of Composites Using Internal Resistive Heating," *ASME Journal of Manufacturing Science and Engineering,* 122(1), 124–131, 2000.
- B. Ramakrishnan and R. Pitchumani, "Fractal Permeation Characteristics of Preforms Used in Liquid Composite Molding," *Polymer Composites,* 21(2), 281–296, 2000
- L. Zhu and R. Pitchumani, "Analysis of a Process for Curing Composites by the Use of Embedded Resistive Heating Elements," *Composites Science and Technology,* 60(14), 2699–2712, 2000.
- F. Yang and R. Pitchumani, "Fractal Description of Interlaminar Contact Development During Thermoplastic Composites Processing," *Journal of Reinforced Plastics & Composites,* 20(7), 536–546, 2001.
- D. R. Nielsen and R. Pitchumani, "Intelligent Model-based Control of Preform Permeation in Liquid Composite Molding Processes, with Online Optimization," *Composites A: Applied Science & Mfg.,* 32(12), 1789–1803, 2001.
- F. Yang and R. Pitchumani, "A Fractal Cantor Set-based Description of Intimate Contact Evolution During Thermoplastic Composites Processing," *Journal of Materials Science,* 36(19), 4661–4671, 2001.
- D. R. Nielsen and R. Pitchumani, "Closed-loop Flow Control in Resin Transfer Molding Using Real-Time Numerical Process Simulations," *Composites Science and Technology,* 62(2), 283–298, 2002.
- F. Yang and R. Pitchumani, "Interlaminar Contact Development in Thermoplastic Fusion Bonding," *Polymer Engineering and Science,* 42(2), 424–438, 2002.
- D. R. Nielsen and R. Pitchumani, "Control of Flow in Resin Transfer Molding with Real-time Preform Permeability Estimation," *Polymer Composites,* 23(6), 2002.

(c) **Books or Book Chapters Submitted and yet to be published:**

1. R. N. Smith, C. Doumanidis, and R. Pitchumani, *Transport Phenomena in Materials Processing*, Chapter in Heat Transfer Handbook, A. Bejan and A. Kraus, eds., Wiley, 2002 (In Press).

(d) **Books or Book Chapters Published:**

1. R. Pitchumani, *Processing of Thermoplastic Composites*, Chapter 4 in Annual Review of Heat Transfer, C. L. Tien, V. Prasad, and F. P. Incropera, eds., Vol. XII, pp. 117–186, Begell House, 2002 (ISBN: 1-56700-166-1)
2. R. Pitchumani, *Transport Phenomena in Materials Processing and Manufacturing*, (with T. L. Bergman, et al.) Vol. HTD-**347**, ASME Press, New York, 1997.
3. R. Pitchumani, *Heat Transfer in Materials Processing*, (with U. Chandra, et al.), Vol. HTD-**361-4**, ASME Press, New York, 1998.
4. R. Pitchumani, *Advances in Sensing and Control for Thermal Processing in Manufacturing*, ASME-HTD CD Volume 0-7918-1997-3, 1463CD, National Heat Transfer Conference Proceedings, 2000.
5. R. Pitchumani, *Proceedings of the ASME Heat Transfer Division – 2000*, with J. H. Kim, et al., Vol. HTD-366-3, ISBN: 0-7918-1908-6, ASME Press, New York, 2000.
6. R. Pitchumani, *Proceedings of the Symposium on Polymer and Composite Materials Processing*, with S.G. Advani and S.T. Holmes, CD-ROM Volume, ASME Press, New York, 2001 (November 2001)

(e) **Printed Technical Reports and Non-refereed Publications:**

1. B. Ramakrishnan, *Studies on Flow and Cure in Resin Transfer Molding Processes*, M.S. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, July 1998.
2. S. K. Padmanabhan, *Stochastic Analysis and Optimization of Liquid Composite Molding Processes*, M.S. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, January 1999.
3. L. Zhu, *Curing of Composites Using Internal Resistive Heating*, M.S. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, August 2000.
4. D. R. Nielsen, *Model Based Control Strategies for Flow in Resin Transfer Molding of Composite Materials*, M.S. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, June 2001.
5. S. B. Streeseman, *Issues Pertaining to Flow in Resin Transfer Molding Processes*, M.S. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, May 2002.
6. F. Yang, *Studies on Interface and Interphase Development in Polymer Matrix Composite Materials*, Ph.D. Thesis (*Advisor: R. Pitchumani*), Department of Mechanical Engineering, University of Connecticut, June/July 2002.
7. P. D. Lafferty and R. Pitchumani, "An Intelligent Model-Predictive Process Control Framework for RTM," in: *Proceedings of the 13th Conference of the American Society for Composites*, A. J. Vizzini, ed., pp. 213–225, 1998
8. F. Yang and R. Pitchumani, "A Fractal Model for Intimate Contact Development During Thermoplastic Fusion Bonding," in: *Proceedings, 13th Technical Conference of the American Society for Composites*, A. J. Vizzini, ed., pp. 1134–1146, 1998

9. L. Zhu and R. Pitchumani, "Processing Envelopes for Supplemental Internal Resistive Heating during Thermosetting Composites Cure," *Proceedings of the Eighth Japan-US Conference on Composites*, G. Newaz and R. Gibson, eds., pp. 122–132, 1998
10. F. Yang and R. Pitchumani, "Modeling Interlaminar Contact Evolution During Thermoplastic Composites Processing Using a Fractal Tow Surface Description," Proceedings *ANTEC 99 Conference,* Society of Plastics Engineers, pp. 1316–1320, 1999
11. D. Nielsen and R. Pitchumani, "Real-Time Model-Predictive Control of Preform Permeation in Liquid Composite Molding Processes," in *Advances for Sensing and Control of Thermal Processing in Manufacturing,* ASME Edited CD Volume, 0-7918-1997-3, 1463CD, National Heat Transfer Conference, Topic Area: T9-35, Paper No. NHTC2000-12158, 10 pp.
12. D. Nielsen and R. Pitchumani, "Control of Flow in Resin Transfer Molding with Real-time Permeability Estimation," in *Proceedings of the Heat Transfer Division – 2000,* ASME HTD-Vol. **366-3**, pp. 159–170, 2000.

(h) <u>Invited Presentations:</u>

1. D. Nielsen and R. Pitchumani, "Intelligent Simulation-based Optimal Control of Liquid Composite Molding Processes," **<u>Invited Presentation</u>**, *On-line Sensing and Control for Liquid Molding of Composite Structures,* Annapolis, MD, April 14–15, 1999
2. R. Pitchumani, "Innovative Strategies for Modeling and Processing of Composite Materials," **Invited Presentation, *Gordon Research Conference on Composites,*** Ventura, CA, January 12, 2000.
3. R. Pitchumani, Invited Presentation, Department of Mechanical Engineering, Columbia University, October 1998.
4. R. Pitchumani, Invited Presentation, Department of Mechanical Engineering, State University of New York, Stony Brook, December 1999.
5. R. Pitchumani, Invited Presentation, Department of Mechanical Engineering, RPI, March 2001.
6. R. Pitchumani, Invited Presentation, Department of Mechanical Engineering, Carnegie-Mellon University, October 2001.
7. R. Pitchumani, Invited Presentation, Department of Mechanical Engineering, Purdue University, April 2002.

(i) <u>Contributed Presentations:</u>

1. P. D. Lafferty and R. Pitchumani, "An Intelligent Model-Predictive Process Control Framework for RTM," Presented at the *13th Conference of the American Society for Composites,* Baltimore MD, 1998.
2. F. Yang and R. Pitchumani, "A Fractal Model for Intimate Contact Development During Thermoplastic Fusion Bonding," Presented at the *13th Technical Conference of the American Society for Composites,* Baltimore MD, 1998.
3. L. Zhu and R. Pitchumani, "Processing Envelopes for Supplemental Internal Resistive Heating during Thermosetting Composites Cure," Presented at the *Eighth Japan-US Conference on Composites*, Baltimore, MD, 1998
4. F. Yang and R. Pitchumani, "Modeling Interlaminar Contact Evolution During Thermoplastic Composites Processing Using a Fractal Tow Surface Description," Presented at the *ANTEC 99 Conference,* Society of Plastics Engineers, New York, 1999
5. D. Nielsen and R. Pitchumani, "Real-Time Model-Predictive Control of Preform Permeation in Liquid Composite Molding Processes," Presented at the National Heat Transfer Conference, ASME, Pittsburgh, PA, 2000.

6. D. Nielsen and R. Pitchumani, "Control of Flow in Resin Transfer Molding with Real-time Permeability Estimation," Presented at the ASME IMECE, Orlando, FL, 2000.

(j) <u>Honors, Awards, or Prizes Received during the Period of the Programs:</u>

1. **OUTSTANDING JUNIOR FACULTY AWARD**, University of Connecticut, Awarded to Dr. Pitchumani, in recognition of *"outstanding scholarly achievements and sustained professional growth,"* 1998.
2. **OUTSTANDING MECHANICAL ENGINEERING FACULTY AWARD,** University of Connecticut, 2000 *In recognition of contributions to the Department in Teaching, Research, and Service*
3. **GUEST EDITOR,** *Journal of Thermoplastic Composite Materials, 1998,* and **EDITORIAL BOARD MEMBER,** *Journal of Thermoplastic Composite Materials,* 1998–
4. **GUEST EDITOR,** *Polymer Composites,* 2002.

(k) <u>Number of Students Supported (at least 1/4 time on ONR funding):</u>

Post-doctoral:	0
Doctoral:	1
Masters:	4
Undergraduate:	1

Of these, the number of

Females:	0
Under-represented Ethnic Groups:	0